

# MECHANICAL CHARACTERIZATION OF SUB-MICROMETER THICK DLC FILMS BY AFM TENSILE TESTING FOR SURFACE MODIFICATION IN MEMS

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## ABSTRACT

This paper describes mechanical properties of Diamond-like Carbon (DLC) films for the protection of MEMS against their friction and wear. The compact tensile tester operated in atomic force microscope (AFM) was developed in this research to evaluate Young's modulus, Poisson's ratio and fracture strength of DLC films. Single crystal silicon (SCS) and DLC coated SCS (DLC/SCS) specimens on a micrometer scale were used in the tests. The DLC films with thickness of 0.2 and 0.3  $\mu\text{m}$  were deposited on 20  $\mu\text{m}$ -thick SCS specimens by Plasma-enhanced Chemical Vapor Deposition (PE-CVD) method of the hot cathode Penning Ionization Gauge (PIG) discharge type. The AFM tensile test and nano-indentation test were revealed that Young's modulus and Poisson's ratio of the DLC films ranged from 99 GPa to 112 GPa, and from 0.36 to 0.46, respectively.

## I. INTRODUCTION

DLC films are one of promising coating materials for MEMS due to its desirable properties of low friction coefficient and high wear resistance [1]. A good understanding of mechanical properties as well as tribological properties of DLC films is required for the improvement of performance and life extension of MEMS devices, since elastic contact between micro scale components plays an important role in surface damage of the devices. So far, elastic properties of DLC films deposited on a substrate have just been estimated by nano-indentation tests since the films are usually possessed of substantial intrinsic stress, which prohibits self-standing of the films. However, the indentation test is not effective for quantitative evaluation of mechanical properties of the films. The indentation test, especially, cannot determine Poisson's ratio and fracture strength of the films. In order to realize mechanical properties of sub-micron thick DLC films for a successful application of the films to MEMS, tensile tests of the films using strain measurement technique [2] are essential.

## II. EXPERIMENTAL PROCEDURE

Tensile testing using AFM (Nanopics NPX100; Seiko

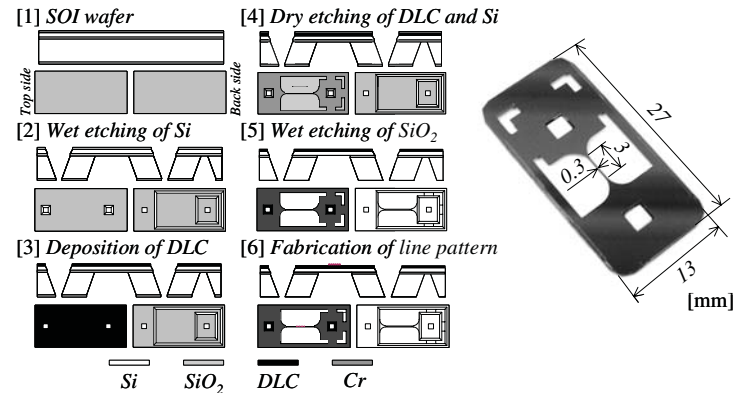
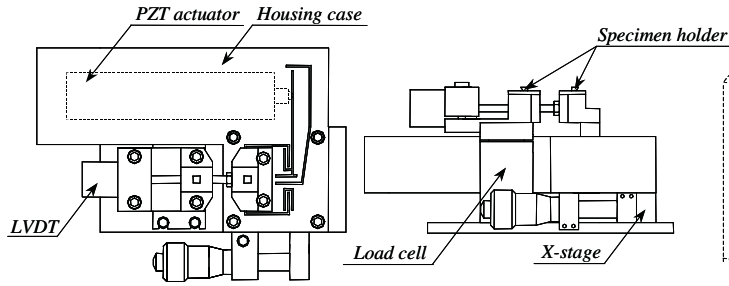


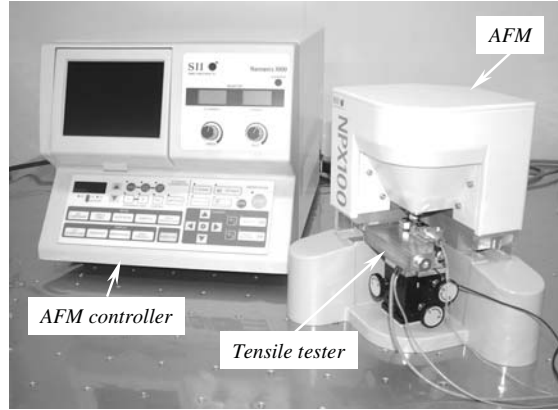
Figure 1 Fabrication process of tensile specimen along with its photograph.

Instruments Inc.) was carried out for 20  $\mu\text{m}$ -thick SCS specimens oriented along the [110] direction in the (001) plane and DLC/SCS specimens. Figure 1 shows the fabrication process of tensile specimen along with its photograph. For making SCS specimens, the deposition and dry etching process of DLC film were excluded. DLC films with sub-micron thickness ranging from 0.20 to 0.33  $\mu\text{m}$  were deposited on SCS specimens at bias voltages of  $-100$  V and  $-300$  V. The resist line pattern was also deposited at the center of specimen for measuring longitudinal and lateral strains using the AFM.

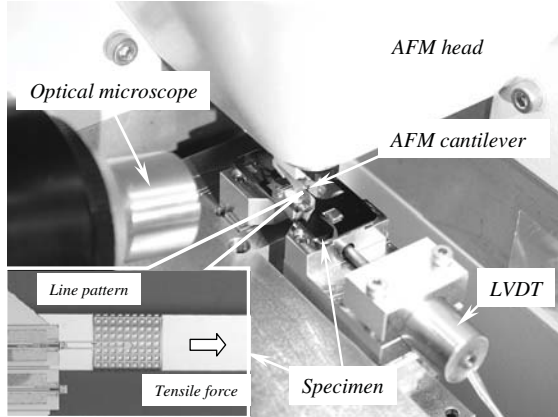
This research has newly developed the compact tensile tester operated in the AFM, as shown in Figs. 2(a) – 2(c). The PZT actuator with a resolution of 1 nm built in the housing case applies tensile force to the specimen. The housing case has a hinge structure that can enlarge two times the stroke in tensile direction of the actuator. The load cell and linear variable displacement transducer (LVDT) measure the tensile force and displacement of the housing case, respectively. The AFM image in Fig. 3 shows the resist line pattern fabricated at the gage section of the DLC/SCS specimen. The AFM can observe the profile of resist lines during the tensile test, and the longitudinal and lateral elongations of the specimen are calculated from the relative displacement between two lines, facing each other. SCS and DLC/SCS specimens are fixed on the tester by hooking up two square holes fabricated in both grip ends of the specimen. After settling the specimen in the tester and braking three supported strips at the end of the grip, the



(a) Schematic of compact tensile tester



(b) Tensile tester built in AFM



(c) Detail of tensile testing

Figures 2 Compact tensile tester operated in AFM.

AFM tensile tests are carried out.

Young's modulus and Poisson's ratio of the SCS specimens were directly derived from the AFM tensile test. Young's modulus of the DLC films was also determined by a resultant modulus,  $E_{1+2}$ , obtained from the tensile tests. The  $E_{1+2}$  is defined as the following equation on the assumption that the tensile strain of DLC film is equal to that of SCS substrate.

$$E_{1+2} = \frac{t_1 E_1 + t_2 E_2}{t_1 + t_2} \quad (1)$$

Here,  $E_1$  and  $t_1$  are Young's modulus and film thickness for

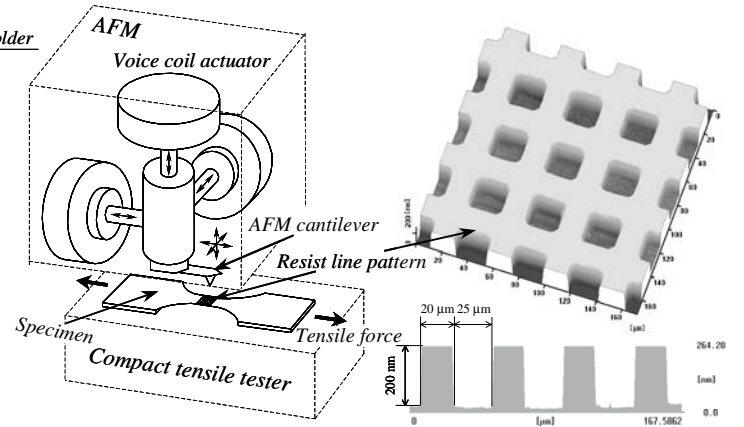


Figure 3 AFM image of resist line pattern fabricated at the gage section of the DLC/SCS specimen.

SCS substrate, and  $E_2$  and  $t_2$  are the same parameters for DLC film. The Young's modulus of DLC film can be calculated if the  $E_{1+2}$  and  $E_1$  are measured in the tensile test.

In order to determine Poisson's ratio for DLC film, not only the tensile test but also a nano-indentation test using a diamond indenter were performed. This is because the SCS substrate restricts the lateral deformation of the film to the tensile axis. This research carried out nano-indentation tests using 2  $\mu\text{m}$ -thick DLC films deposited on a SCS substrate, following the test procedure proposed by Oliver, et al [3]. In the nano-indentation test, the unloading stiffness,  $S$ , is obtained from the gradient of the unloading curve in the load-displacement relation. The unloading stiffness is formulated as;

$$\frac{1}{S} = C_f + \frac{\sqrt{\pi}}{2E_r} \frac{1}{\sqrt{A}} \quad (2)$$

where,  $C_f$  is the compliance of load frame,  $E_r$  is the reduced modulus, and  $A$  is the contact area of the indenter. The compliance of load frame must be calibrated prior to the test. The reduced modulus,  $E_r$ , is also expressed by the sum of plane-strain moduli of the DLC film and the indenter as the following equation.

$$\frac{1}{E_r} = \frac{(1-\nu_2^2)}{E_2} + \frac{(1-\nu_i^2)}{E_i} \quad (3)$$

Here,  $\nu_2$  is Poisson's ratio of the DLC film.  $E_i$  and  $\nu_i$  are Young's modulus and Poisson's ratio for the indenter. If the  $E_r$  experimentally determined in the nano-indentation test, the Poisson's ratio,  $\nu_2$ , of the DLC film is calculated by applying the Young's modulus of the film and plane-strain modulus of the indenter to Eq. (3).

### III. RESULTS AND DISCUSSIONS

Figure 4 shows typical tensile stress-strain relations of

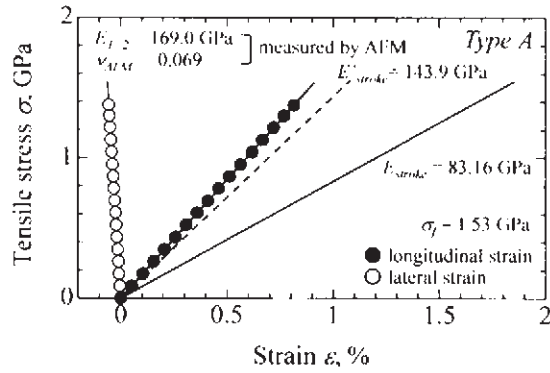


Figure 4 Typical tensile stress-strain relations of SCS specimen until its fracture.

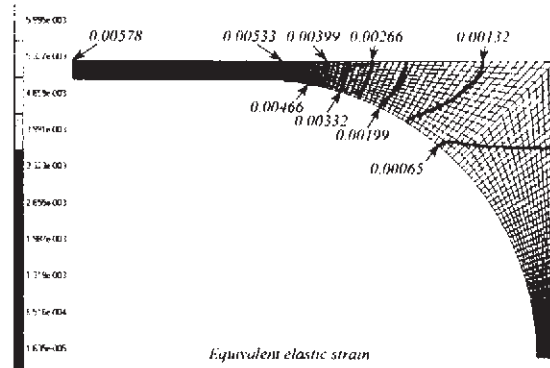
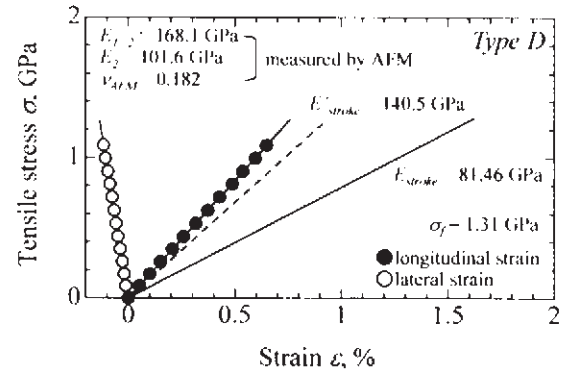


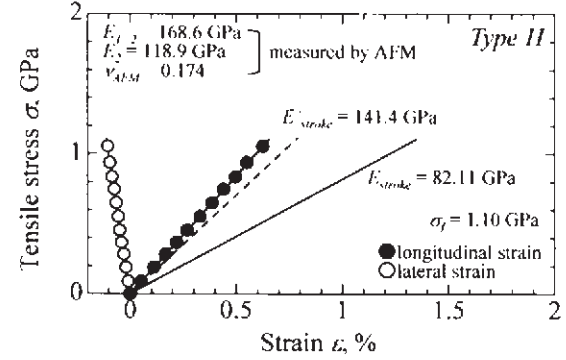
Figure 5 Stress distribution in specimen derived from FEA.

SCS specimen until its fracture. Closed and open plots indicate longitudinal and lateral strains measured by the AFM, respectively. The solid line corresponds to the strain estimated by the stroke of the housing case, which obtained from the LVDT. The dashed line shows the revised strain of the solid line data by finite element analyses (FEA) shown in Fig. 5. The FEA calculated the elongation at the gage section of the specimen, and it revealed that the elongation at the gauge was 0.58 times of that of the specimen length. Young's modulus of the SCS specimen derived from the AFM measurement is 169.0 GPa, which is in a good agreement with analytical value based on the anisotropic theory. Poisson's ratio of the SCS is 0.069, which also agree with analytical value, 0.064. The fracture strength, 1.53 GPa, is identical to that of other works [4]. However, the Young's modulus calculated from the stroke of housing case is 83.16 GPa, which is very small. Even the Young's modulus revised by the FEA indicates 143.9 GPa, which is 17 % smaller than the analytical value. Therefore, the AFM tensile test can precisely evaluate mechanical properties of a micro-scaled self-standing sample.

Figures 6(a) and 6(b) represent stress-strain relations of DLC/SCS specimens with the film thickness of 0.32  $\mu\text{m}$  and 0.21  $\mu\text{m}$ , respectively. In Fig. 6(a), the resultant modulus of the DLC/SCS specimen deposited at -100 V shows 168.1 GPa, which is close to the Young's modulus of



(a)  $B_v = 100 \text{ V}$



(b)  $B_v = 300 \text{ V}$

Figures 6 Stress-strain relations of DLC/SCS specimens with the film thickness of (a) 0.32  $\mu\text{m}$  and (b) 0.21  $\mu\text{m}$ .

SCS substrate. Inserting the resultant modulus, the Young's modulus of SCS substrate and thickness of the film and substrate into Eq. (1), we have found that the Young's modulus of DLC film is 101.6 GPa. The resultant modulus of the DLC/SCS specimen deposited at -300 V is 168.6 GPa in Fig. 6(b), which have led the Young's modulus of the film to be 118.9 GPa. Poisson's ratio of the DLC film in Fig. 6(a) and 6(b) obtained from the AFM measurement ranged from 0.167 to 0.182. However, these results are deferent from real value of the DLC film since the lateral strain of SCS substrate is not equal to that of the film. In this research, the Poisson's ratio of the DLC films was calculated from plane-strain modulus obtained in nano-indentation tests.

Table 1 summarizes the Young's modulus, Poisson's ratio and fracture strength of the SCS and DLC/SCS specimens. Young's moduli of DLC films deposited at -100 V and -300 V averaged 99.05 GPa and 111.7 GPa, respectively. The Young's modulus obtained from the tensile test is slightly smaller than the reduced modulus in the nano-indentation test. The Poisson's ratio of DLC film deposited at -100 V indicates 0.362, which is quite different from that measured at the top surface of DLC/SCS specimen by the AFM. Since the Poisson's ratio of SCS substrate in the [110] direction in the (001) plane is very small, the lateral deformation of DLC film has not been produced in the tensile test. The Poisson's ratio of DLC film deposited at -300 V also shows the lager value, 0.461.

Table 1 Summarizations of Young's modulus, Poisson's ratio and fracture strength of SCS and DLC/SCS specimens.

Specimen	Bias voltage $B_1$ [V]	Thickness		AFM tensile test			Indentation test		Fracture strength	
		DLC $t_2$ [ $\mu\text{m}$ ]	SCS $t_1$ [ $\mu\text{m}$ ]	Young's moduli		Poisson's ratio $\nu_{AFM}$	Reduced modulus $E_r$ [GPa]	Poisson's ratio $\nu_2$	DLC/SCS $\sigma_f$ [GPa]	DLC $\sigma_2$ [GPa]
SCS	A	—	19.0	169.0 ( $E_f$ )	—	0.0694	—	—	1.53	—
	B	—	19.0	169.2 ( $E_f$ )	—	0.0701	—	—	1.36	—
	C	—	19.0	168.8 ( $E_f$ )	—	0.0691	—	—	1.49	—
	Ave.	—	19.0	169.0 ( $E_f$ )	—	0.0695	—	—	1.46	—
DLC/SCS	D	0.320	19.0	168.1	101.6	0.182	—	0.335	1.31	0.793
	E	0.330	19.0	168.0	103.0	0.176	104.7	0.315	1.22	0.748
	F	0.300	19.0	168.0	92.61	0.177	—	0.436	1.29	0.712
	Ave.	0.317	19.0	168.0	99.05	0.178	—	0.362	1.27	0.751
	G	0.200	19.0	168.7	119.3	0.167	—	0.407	1.16	0.819
	H	0.210	19.0	168.6	118.9	0.174	128.1	0.410	1.10	0.774
	I	0.240	19.0	168.3	97.02	0.172	—	0.567	0.990	0.571
	Ave.	0.217	19.0	168.5	111.7	0.171	—	0.461	1.08	0.722

than that derived from the AFM measurement. Then, the evaluation technique of Young's modulus and Poisson's ratio proposed here is efficient for thin films coated on a specimen.

The fracture strength of DLC/SCS specimens is smaller than that of the SCS specimen obtained in Fig. 5. The reduction of the strength is probably caused by nucleation and propagation of crack at the film surface. The crack of the film could have induced a surface defect of the SCS substrate, so that SCS substrate fractured in brittle manner as depicted in Fig. 7. However, the essences of the fracture mechanism of DLC film cannot be concluded until the fracture surface have been observed.

#### IV. CONCLUSION

We developed the AFM tensile tester for mechanical characterization of the SCS specimens and sub-micrometer thick DLC films. The AFM tensile test could precisely evaluate mechanical properties of the micro-scaled self-standing SCS specimens. The tensile test using with nano-indentation test were also effective for mechanical characterization of the DLC films deposited on the SCS specimen. The Young's modulus of the DLC films deposited at  $-100$  V and  $-300$  V averaged 99.05 GPa and 111.7 GPa, respectively. The Poisson's ratio of the films evaluated from the AFM tensile test and the nano-indentation test, were 0.362 for the bias voltage of  $-100$  V and 0.461 for  $-300$  V. This research for the first time showed the fracture strength of the DLC films ranged from 0.571 GPa to 0.819 GPa.

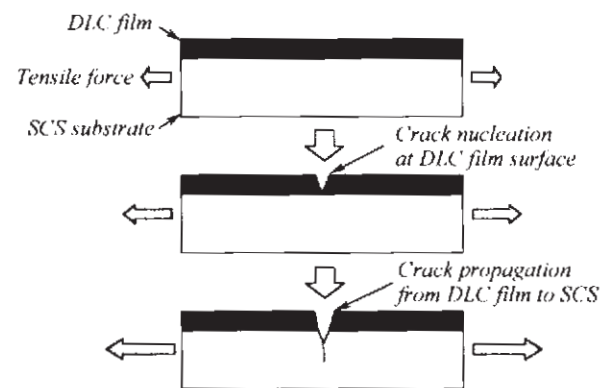


Figure 7 Fracture mechanism of DLC/SCS tensile specimen.

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